भारतीय मानक
Indian Standard

IS 644 : 2023

रंग रोगन के लिए डाईपेन्टीन — विशिष्टि

(तीसरा पुनरीक्षण)

Dipentene for Paints — Specification

(Third Revision)

ICS 87.060.10

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June 2023 **Price Group 6**

Raw Materials for Paints, Varnishes, and Related Product Sectional Committee, CHD 21

FOREWORD

This Indian Standard (Third Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Raw materials for paints, varnishes and related product Sectional Committee had been approved by the Chemical Division Council.

This Indian Standard was first published in 1955 based on data available for imported dipentene. However, the indigenous production of dipentene commenced in the early 1960's. Consequently, there is practically no import of dipentene since 1960. This standard was therefore revised in 1969 in order to take care of indigenous technology. In the first revision, requirements for distillation range and flash point had been modified. Additional requirements for residue on evaporation, copper corrosion, unpolymerizable matter, grease spot test, and kauri-butanol value were included. Subsequently, the second revision was done in 1992, in which, requirements for flash point, residue on evaporation, refractive index, relative density, distillation range, and unpolymerizable matter had been modified and new requirement ash content had been added. Further, the temperature of reporting for relative density and refractive index had been changed from 30 °C to 27 °C.

This revision has been taken to bring out the standard in the latest style and format of the Indian Standard. It also incorporates amendments issued to the last version of the standard. In addition, the following changes have been made:

a) The existing qualitative test method of potassium dichromate solution for colour comparison has been replaced with the quantitative method of hazen unit scale and the requirement of colour has been modified to maximum 50 hazen unit;

b) The recommended density range has been increased from 0.843 to 0.855 to 0.843 to 0.860 keeping in mind that dipentene is a mixture of solvents derived from natural sources. The density keeps changing depending upon the nature of pine tree and other natural sources;

c) The minimum temperature for distillation range has been reduced from 170 °C to 160 °C;

d) The requirement of unpolymerizable matter has been modified to unpoloymerizable matter or matter insoluble in sulphuric acid in order to cover all types of impurities in the dipentene. Further, the maximum limit of impurities has been reduced from 15 percent by volume to 4 percent by volume;

e) The requirement of kauri-butanol value has been deleted as kauri-resin is not readily available in the market and other prescribed requirements are taking care of the required needs of the product; and

f) Considering, the adverse impact of lead and heavy metals on human health, the maximum limits for lead and heavy metals have been prescribed.

The committee responsible for formulation of this standard is listed in Annex H.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

DIPENTENE FOR PAINTS — SPECIFICATION

(Third Revision)

1 SCOPE

This standard prescribes the requirements and the methods of sampling and test for dipentene used as a thinner and solvent for paints.

2 REFERENCES

The standards listed in Annex A contain provisions which, through reference in this text constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated in Annex A.

3 TERMINOLOGY

3.1 For the purpose of this standard, the definitions given under **2** of IS 82, IS 1303 and the following shall apply.

3.2 Registered Sample

Sample supplied in advance by a prospective supplier and registered by the approved testing authorities after testing it to all the requirements of this standard. A complete record of its performance shall be kept in respect of all tests.

3.3 Approved Sample

The sample accepted by the indentor or inspection authority as the basis for supply. When a sample is tested and approved by the purchaser or inspection authority, the result of such tests as permits the supplier to meet the limits imposed by the specification for deliveries shall be made available to the supplier.

4 REQUIREMENTS

4.1 Description

The material shall be a mixture of monocyclic terpenes, namely, Alpha terpinene, Limonene, Gamma terpinene, and Terpinolene; bicyclic terpenes, namely, Car-3-ene and Para-cymene with characteristic odour. It shall be clear and free from sediment, suspended matter, and undissolved water.

4.2 Lead Restriction

The material shall be tested for restriction from lead in accordance with IS 101 (Part 8/Sec 5), or by atomic absorption spectrometer, or by Inductively coupled plasma (ICP) spectroscopy. When thus tested the material shall not contain lead or compounds of lead or mixtures of both, calculated as metallic lead exceeding 90 ppm (*see* note).

NOTE — When no lead is used during production, the product is considered lead free.

4.3 Toxic Heavy Metal Restriction

Product shall not be manufactured using mercury and mercury compounds, cadmium, chromium VI, arsenic, antimony, and their oxides. The material shall not contain more than 0.1 percent by weight in total of above toxic heavy metals in the form of natural impurities or impurities entailed by the production process which are contained in the raw material when tested by the relevant Atomic Absorption Spectroscopic methods.

4.4 The material shall also comply with the requirements given in Table 1.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in suitable containers as agreed to between the purchaser and the manufacturer.

5.2 Marking

5.2.1 The packing shall be securely closed and marked with the following particulars:

a) Name of the material;
b) Manufacturer's name and trade-mark, if any;
c) Mass of the material;
d) Lead content (Maximum);
e) Toxic heavy metals content;
f) Batch number or Lot number in code or otherwise;
g) Month and year of manufacture; and
h) A cautionary note as below:
    1) Keep out of reach of children, or
    2) This product may be harmful if swallowed or inhaled.

5.2.2 *BIS Certification Marking*

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act*, 2016 and the Rules and Regulations framed thereunder, and the product(s) may be marked with the Standard Mark.

Table 1 Requirements for Dipentene for Paints

(*Clause* 4.4)

Sl No.	**Characteristic**	**Requirement**	**Method of Test, Ref to**	
			Annex	Indian Standard
(1)	(2)	(3)	(4)	(5)
i)	Colour, in Hazen scale, *Max*	50	B	-
ii)	Grease spot test	To pass the test	C	-
iii)	Flash point, °C, *Min*	35	-	IS 354 (Part 1)
iv)	Ash, percent by mass, *Max*	0.01	D	-
v)	Residue on evaporation, percent by mass, *Max*	2.00	-	IS 82
vi)	Refractive index at 27 °C (*see* note 1)	1.470 to 1.480	-	IS 354 (Part 1)
vii)	Relative density, 27°/27 °C (*see* note 2)	0.843 to 0.860	-	IS 82
viii)	Distillation range, Initial b.p., for 95 ml	*Min* 160 °C *Max* 195 °C	-	IS 82
ix)	Unpolymerizable matter or matter insoluble in sulphuric acid, percent by volume, *Max*	4	E	-
x)	Acid value, *Max*	1.0	F	-
xi)	Corrosive Sulphur (Copper corrosion)	Shall not blacken or corrode clean copper strip	-	IS 82

NOTE

1 The refractive index determined at any temperature within the range 20 °C to 30 °C can be adjusted to 27 °C using the correction factor of ± 0.000 46 for every degree Celsius fall and rise of temperature

2 The relative density is synonymous with specific gravity. It may be determined at any temperature within the range of 20 °C to 30 °C and can be adjusted to 27°/27 °C using the correction factor of 0.000 8 for every degree Celsius fall and rise of temperature.

ANNEX A

(*Clause* 2)

LIST OF REFERRED INDIAN STANDARDS

IS No.	*Title*
IS 82 : 1973	Method of sampling and test for thinners and solvent for paints (*first revision*)
IS 101 (Part 8/Sec 5) : 2022	Methods of sampling and test for paints, varnishes and related products: Part 8 Tests for pigments and other solids, Sec 5 Lead restriction test (*fourth revision*)
IS 266 : 1993	Sulphuric acid — Specification (*third revision*)
IS 323 : 2009	Rectified spirit for industrial use — Specification (*second revision*)
IS 354 (Part 1) : 1987	Method of sampling and test for resins for paints: Part 1 General test method (*second revision*)
IS 361 : 2009	Normal butyl alcohol, technical — Specification (*third revision*)
IS 537 : 2011	Toluene — Specification (*second revision*)
IS 878 : 2008	Laboratory glassware — Graduated measuring cylinders (*second revision*)
IS 1070 : 1992	Reagent grade water — Specification (*third revision*)
IS 1303 : 1983	Glossary of terms relating to paints (*second revision*)
IS 1997 : 2008	Laboratory glassware — Burettes (*third revision*)
IS 2837 (Part 2) : 1977	Specification for porcelain crucibles and basins: Part 2 Basins (*first revision*)
IS 4905 : 2015	Random sampling and randomization procedures (*first revision*)

ANNEX B

[*Table* 1, *Item* (i)]

DETERMINATION OF COLOUR IN APHA SCALE

B-1 PRINCIPLE

The color of a liquid sample is measured using an instrument capable of measuring transmitted colour and reporting in Pt-Co colours (APHA Scale) or in a colour system that can be converted into Pt-Co colours.

B-2 APPARATUS AND MATERIALS

B-2.1 Colour-measuring instrument, spectrophotometer capable of measuring transmitted colour (0°/180° geometry) and reporting the results in the Pt-Co colour scale.

B-2.2 Absorption cells, 50 mm light path length recommended, unless a different path length is specified by the instrument manufacturer.

B-3 PROCEDURE

Baseline calibration of the instrument shall be performed following the instrument manufacturer’s recommendations.

If the material shows any visual haziness, remove the haze by using suitable processes such as filtration, centrifugation, heating, ultrasonic treatment or any other suitable means. Ensure that the sample is not changing colour during the treatment to remove haze. If the haziness cannot be removed, the measured value will be unreliable.

Then, using the same type of absorption cell as used for the baseline calibration, fill the absorption cell with the product. Take care not to touch the measurement area of the glass tube or absorption cell. Avoid creating air bubbles when filling the glass tube or absorption cell. If air bubbles are formed and remain trapped, remove them by heating, vacuum, ultrasonic treatment or any other suitable means.

Insert the absorption cell in the instrument and measure the Pt-Co color, following the instrument manufacturer’s recommended procedure. Regular checks as per the instrument manufacturer’s recommendations should be carried out. These will normally be in the form of checks with certified reference materials.

Report the color in Pt-Co color (APHA) units as given by the instrument.

ANNEX C

[*Table* 1, *Item* (ii)]

GREASE SPOT TEST

C-1 GENERAL

C-1.1 Outline of the Method

A few drops of the material is evaporated on a filter paper and observed for any oily spot on it.

C-2 Apparatus

C-2.1 Porcelain Basin

Evaporating, with spout, flat bottom, 70 mm in diameter [*see* IS 2837 (Part 2)].

C-3 PROCEDURE

Place 5 drops of the material by means of a small pipette at the centre of a clean white filter paper supported on the porcelain basin and allow the liquid to evaporate at room temperature, away from direct sunlight. Examine after 30 min whether there is any oily or greasy spot on the filter paper.

C-4 REPORT

The material shall be deemed to have passed the test if no oily or greasy spot is seen on the filter paper.

ANNEX D

[*Table* 1, *Item* (iv)]

DETERMINATION OF ASH

D-1 GENERAL

D-1.1 Outline of the Method

Ash content is determined by igniting the material, under specified conditions and weighing the non-combustible material.

D-2 APPARATUS

D-2.1 Porcelain Crucible or Dish

D-2.2 Muffle Furnace - Range up to 1 200 °C.

D-3 PROCEDURE

Weigh a clean and previously ignited porcelain crucible or dish, accurately to the nearest 0.000 1 g. Add 10 ml to 25 ml of the material and weigh again. Heat the material gently by bringing a flame on the bottom and sides of the crucible until the oil ignites. Reduce the size of the flame until the heat is just sufficient to keep the material burning. Continue heating of the sample to a black char and transfer it to a muffle furnace or heat it over a burner until all carbonaceous matter is reduced to ash. Remove the crucible, place in a desiccator and cool to room temperature. Weigh the repeat heating and cooling until the difference between two weighings is less than 1 mg.

D-4 RESULTS

The material shall be declared to have passed the test if the ash content does not exceed 0.01 percent by mass.

ANNEX E

[*Table* 1, *Item* (ix)]

DETERMINATION OF UNPOLYMERIZABLE MATTER OR MATTER INSOLUBLE IN SULPHURIC ACID

E-1 GENERAL

E-1.1 Outline of the Method

The amount of unpolymerizable matter left after action of sulphuric acid of specified concentration on the material is determined,

E-2 APPARATUS

E-2.1 Measuring Cylinder

Stoppered, of 50 ml capacity (*see* IS 878).

E-3 REAGENT

E-3.1 Concentrated Sulphuric Acid, 98 percent (*w/w*) (*see* IS 266).

E-4 PROCEDURE

Add exactly 10 ml of the material in drops to 20 ml of sulphuric acid contained in the 50 ml cylinder immersed in ice water and keep the cylinder in ice water for about 15 minutes. Shake the cylinder vigorously during addition of the material. Remove the cylinder from ice water, shake well, immerse in a water-bath and heat at 60 °C to 65 °C for about 10 minutes with frequent shaking of the contents. Return the cylinder to cold water-bath to attain room temperature. Add sufficient amount of sulphuric acid to bring the level of the liquid of the upper layer into the middle and calculate the unpolymerized matter as percentage by volume of the original sample.

ANNEX F

[*Table* 1, *Item* (x)]

DETERMINATION OF ACID VALUE

F-l APPARATUS

F-l.1 Conical Flask, 100 ml to 250 ml capacity.

F-l.2 Measuring Cylinder, 25 ml capacity.

F-l.3 Burette, 25 ml capacity graduated in lengths of a millilitre (*see* IS 1997).

F-2 REAGENTS

F-2.1 Ethyl Alcohol, 95 percent by volume, or rectified spirit (*see* IS 323). To remove impurities which might get coloured by potassium hydroxide solution and interfere with the sharpness of the end point, reflux the 95 percent alcohol or rectified spirit over solid sodium hydroxide for 6 h to 8 h, and then distil.

F-2.2 Potassium Hydroxide Solution, 0.1 N approximately.

F-2.3 Phenolphthalein Indicator

Dissolve 0.1 g of phenolphthalein in 100 ml of 60 percent rectified spirit.

F-2.4 Phenol Red

Dissolve 0.1 g of phenol red in 100 ml of rectified spirit.

F-3 PROCEDURE

F-3.1 Weigh about 2.5 g of dried material and dissolve in 20 ml of rectified spirit (**F-2.1**) previously neutralized to phenolphthalein indicator with potassium hydroxide solution. Add about 10 drops of indicator except in the case of phenolic perfumery material and titrate with potassium hydroxide solution until the solution remains fantly pink after 10 seconds of shaking. The flask and its content may be retained for the determination of ester value. If the perfumery material under examination contains phenols or compound with phenolic groups, use phenol red as indicator instead of phenolphthalein.

F-3.2 Calculation

$$\text{Acid value} = \frac{56.1 \times V \times \text{N}}{M}$$

where

V = volume, in ml, of standard potassium hydroxide solution;

N = normality of the potassium hydroxide solution; and

M = mass, in g, of the material taken for the test.

NOTES

1 Sometimes, certain samples of perfumery materials, such as oils of vetiver and geranium, have very high acid values (20 and above). In such case 2.5 g of the material requires even more than 10 ml of 0.1 N aqueous potassium hydroxide solution. With highly acidic materials, it is recommended that 0.75 g to 1 g of the material be taken instead of 2.5 g.

2 In case of benzyl formate which may get hydrolyzed under the above condition, it is desirable to cool the sample below 5 °C before titration.

ANNEX G

(*Clause* 6)

SAMPLING OF DIPENTENE FOR PAINTS

G-l SAMPLING

G-l.1 General Requirements

While drawing samples, the following precautions and directions should be adhered to.

G-1.1.1 Samples shall not be taken in an exposed place.

G-1.1.2 The sampling apparatus shall be clean and dry when used.

G-1.1.3 The samples shall be placed in clean, dry and airtight containers made of suitable material on which dipentene has no action.

G-1.1.4 Once the container is opened, there is a risk of deterioration of contents due to the presence of air. Care should be taken to avoid risk of polymerization of the sample by undue exposure to air or strong light.

G-1.1.5 After filling, the sample containers shall be marked with all details of sampling.

G-2 SCALE OF SAMPLING

G-2.1 Lot

All the containers of the same capacity containing dipentene produced under similar condition of manufacture shall constitute a lot. If the material is supplied in bulk in drums of capacity 500 litres or more, each drum shall constitute a lot.

G-2.1.1 For ascertaining the conformity of the lot to the requirement of the specification, tests shall be carried out for each lot separately.

G-2.2 In the case of containers of capacity less than 500 litres, the number of containers selected from each lot shall depend on the size of the lot and shall be in accordance with Table 2.

G-2.3 These containers shall be selected at random from the lot as per IS 4905.

Table 2 Scale of Sampling
(*Clause* G-2.2)

Sl No.	Lot Size	Number of Containers to be Selected
(1)	(2)	(3)
i)	up to 25	2
ii)	26 to 50	3
iii)	51 to 100	5
iv)	101 to 300	8
v)	301 and above	13

G-3 PREPARATION OF TEST SAMPLES

G-3.1 From each of the selected containers a representative portion of the material shall be taken as per **G-3.2** and **G-3.3**, which shall be sufficient for carrying out tests for all the characteristics given in the specification.

G-3.2 Individual Sample

G-3.2.1 A sample of 200 ml of the liquid shall be withdrawn from each selected containers as per **G-3.1** and kept in separate sample containers of such size that they are nearly filled by the sample. These are termed as individual' samples.

G-3.2.2 When the sample is supplied in drums of capacity 500 litres or more, samples shall be collected 7 from each drum at five different depths with the help of a suitable sampling instrument. These five samples of 200 ml each, shall be kept in separate sample containers of such size that they are nearly filled by the sample.

G-3.3 Composite Sample

Out of the portions (*see* **G-3.2**), a small but equal quantity of the material shall be taken as mixed thoroughly to form a composite sample. In the case of material supplied in drums, the composite sample shall be prepared by mixing thoroughly the portions collected at different depths.

G-4 NUMBER OF TESTS

Each of the individual samples shall be examined for the colour of the solvent and Kauri-butanol value. The test for all other characteristics shall be conducted on composite sample.

G-5 CRITERIA FOR CONFORMITY

The lot shall be deemed to conform to the specification if all the test results according to **G-4** satisfy the corresponding requirement given in Table 1.

ANNEX H
(*Foreword***)**

COMMITTEE COMPOSITION

Raw materials for Paints, Varnishes and Related Products Sectional Committee**,** CHD 21

Organization	*Representative(s)*
In Personal Capacity (Retired from IIT Bombay)	PROF A. S. KHANNA **(*Chairperson*)**
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